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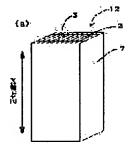
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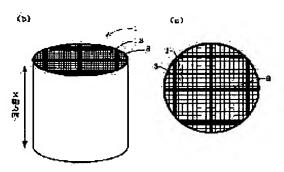
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# (54) HONEYCOMB FILTER

# (57)Abstract:

PROBLEM TO BE SOLVED: To provide a honeycomb filter which can suppress excess increase in temperature and has excellent durability with little irregular temperature distribution. SOLUTION: The honeycomb filter 1 is produced by joining and integrating a plurality of honeycomb segments 12 with a joining material 8, each segment having many passing holes 3 separated by partition 2 and penetrating in the axial direction. The ratio  $\kappa s/\kappa a$  of the thermal conductivity  $\kappa s$  of the honeycomb segment 12 to the thermal conductivity  $\kappa a$  of the joining material 8 ranges from 5 to 300. The density  $\rho a$  of the joining material 8 ranges from 0.1 to 4 g/cc.





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### **CLAIMS**

# [Claim(s)]

[Claim 1] The honeycomb filter characterized by being in the range two or more honeycomb segments of whose which have the circulation hole of a large number penetrated to shaft orientations divided by the septum are the honeycomb filters which come to carry out junction unification through a jointing material for corrugated fibreboard, the ratio of thermal conductivity kappas of said honeycomb segment to thermal conductivity kappaa of said jointing material for corrugated fibreboard and whose kappa s/kappa a are within the limits of 5-300 and, whose consistency rhoa of said jointing material for corrugated fibreboard is 0.1-4g/cc.

[Claim 2] The honeycomb filter according to claim 1 with which the heat capacity Ha per [ which is expressed with specific heat Cpax consistency rhoa of said jointing material for corrugated fibreboard ] unit volume is characterized by being in the range of 0.1x106 - 3x106 J/m3 and K.

[Claim 3] The honeycomb filter according to claim 1 or 2 characterized by a jointing material for corrugated fibreboard having pore.

[Claim 4] A honeycomb filter given in claim 1 characterized by a jointing material for corrugated fibreboard containing a metal thru/or any 1 term of 3.

[Claim 5] A honeycomb filter given in claim 1 characterized by including two or more jointing materials for corrugated fibreboard with which both [ one side or ] thermal conductivity kappaa and unit volume differ thru/or any 1 term of 4. [ of heat capacity Ha ] [ both ]

[Claim 6] A honeycomb filter given in claim 1 to which coefficient of thermal expansion of a jointing material for corrugated fibreboard is characterized by being the range of 1x10-6 - 8x10-6/degree C thru/or any 1 term of 5.

[Claim 7] A honeycomb filter given in claim 1 to which a honeycomb segment is characterized by using silicon carbide or silicon-silicon carbide composite material as a principal component thru/or any 1 term of 6.

[Claim 8] A honeycomb filter given in claim 1 characterized by carrying out the closure of the opening of the predetermined circulation hole in a honeycomb segment in the end face of 1, and carrying out the closure of the opening of a residual circulation hole in other end faces thru/or any 1 term of 7.

[Claim 9] A honeycomb filter given in claim 1 to which more than 70 capacity % of a honeycomb filter is characterized by consisting of 900mm honeycomb segments it is [ segments ] 2 2-10000mm by the cross section thru/or any 1 term of 8.

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### **DETAILED DESCRIPTION**

# [Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention can control too much rise of especially temperature about the honeycomb filter used for the particle uptake filter in exhaust gas, such as an internal combustion engine and a boiler, etc., and dispersion in temperature distribution is related with the honeycomb filter which was excellent in endurance few.

[0002]

[Description of the Prior Art] The honeycomb filter is used for the uptake filter of the particle in exhaust gas, such as an internal combustion engine and a boiler, especially a diesel particle etc. [0003] The honeycomb filter used for such the purpose has the structure where while becomes the opposite side mutually and the closure of the adjoining circulation hole 3 was generally carried out at the end so that it might have the circulation hole 3 of a large number penetrated to X shaft orientations divided by the septum 2 and an end face might present the shape of a checker, as shown in drawing 6. In the honeycomb filter which has such structure, a processed fluid is discharged from the circulation hole 3 with which it flows into the circulation hole 3 3 with which the closure of the input side edge side 42 is not carried out. i.e., the circulation hole with which the closure of the tap hole side edge side 44 is carried out, the closure of the next circulation hole 3 42, i.e., input side edge side, is carried out through the porous septum 2, and the closure of the tap hole side edge side 44 is not carried out. Under the present circumstances, a septum 2 serves as a filter, for example, the soot (soot) discharged from a diesel power plant is caught by the septum, and it deposits on a septum. Thus, the temperature distribution within honeycomb structure became uneven by the temperature change with rapid exhaust gas, or local generation of heat, and the honeycomb filter used had problems, such as producing a crack in a honeycomb filter. When used as a filter (it is called Following DPF) which carries out uptake of the particulate matter under exhaust air of a diesel power plant especially, the collected carbon particle was burned, to remove and reproduce is required, local elevated-temperatureization started on this occasion, and there was a problem of being easy to generate the decline in regeneration efficiency by ununiformity-izing of regenerating temperature and the crack by big thermal stress. Moreover, since the temperature distribution at the time of playback were not uniform, it was difficult to consider as optimum temperature over the whole filter, and it difficult to aim at improvement in regeneration efficiency.

[0004] For this reason, the approach of joining the segment which divided the honeycomb filter into plurality with a jointing material for corrugated fibreboard was proposed. For example, the manufacture approach of the honeycomb structure object which joins much honeycomb objects to a U.S. Pat. No. 4335783 official report with a discontinuous jointing material for corrugated fibreboard is indicated. Moreover, after carrying out extrusion molding of the matrix segment of the honeycomb structure which consists of a ceramic ingredient to JP,61-51240,B, processing the periphery section after baking and making it smooth, it is substantially [ as a matrix segment ] the same, and the thermal-shock-resistance rotation accumulation type at which the difference of coefficient of thermal expansion applies and calcinates [ the mineral composition after calcinating to the joint ] the ceramic jointing material for corrugated fibreboard which becomes with 0.1% or less in 800 degrees C is propose. Moreover, the ceramic honeycomb structure object which similarly joined the honeycomb segment of cordierite to the SAE paper 860008 in 1986 into cordierite cement is indicated. The ceramic honeycomb structure object which furthermore pasted up the honeycomb ceramic member on JP,8-28246,A by the nature sealant of elasticity which consists of the inorganic fiber which is each other interwoven with in three dimensions at least, an inorganic binder, an organic binder, and an inorganic particle is indicated. Moreover, for thermal conductivity to be high, to

prevent local elevated-temperature-ization by making a honeycomb filter using the ingredient of a heat-resistant high silicon carbide system etc., and to prevent breakage of the honeycomb filter by thermal stress is also tried.

[0005] however, the thing to segment -- and/or, although the breakage by thermal stress could be controlled to some extent by using a heat-resistant high ingredient like the ingredient of a silicon carbide system, the temperature gradient of the periphery section of a honeycomb filter and a core could not be canceled, but it was inadequate in respect of improvement in the endurance by uniform playback. Moreover, also when local generation of heat at the time of playback arose, it was.

[0006] Moreover, the thickness of a sealant (jointing material for corrugated fibreboard) layer is 0.3-5mm, and the filter which equalizes the whole temperature and a partial cinder cannot produce easily due to considering as the ceramic filter aggregate of the thermal conductivity 0.1 - 10 W/mk is indicated by the JP,2001-162119, A official report. however, the temperature gradient generated when what can lose a partial cinder and can gather the regeneration efficiency of a soot by making the thickness and the thermal conductivity of a jointing material for corrugated fibreboard into the fixed range carries out elevatedtemperature generation of heat locally -- controlling -- thermal stress -- stopping -- not enough -- a soot -- in respect of improvement in the refreshable amount of marginal soots, it was inadequate. Moreover, although the thermal conductivity and heat capacity of a jointing material for corrugated fibreboard can be adjusted by changing the thickness of a jointing material for corrugated fibreboard as indicated by this official report Since another fault that decrease the effective area of a filter and the property of pressure loss with a soot falls will be produced if the thickness of a jointing material for corrugated fibreboard is increased, When it is going to control the pressure loss of low-fever conductivity, high temperature capacity, and a filter by thickness of a jointing material for corrugated fibreboard, it becomes a rebellion property and there is a limitation in seal thickness actually applicable to a filter. [0007]

[Problem(s) to be Solved by the Invention] This invention is made in view of such a situation, even if especially the place made into the purpose does not change the thickness of a jointing material for corrugated fibreboard, too much rise of temperature can be controlled, and it is in offering the honeycomb filter excellent in endurance.

[8000]

[Means for Solving the Problem] This invention is a honeycomb filter with which it comes to carry out the junction unification of two or more honeycomb segments which have the circulation hole of a large number penetrated to shaft orientations divided by the septum through a jointing material for corrugated fibreboard. The honeycomb filter characterized by being in the range the ratio of thermal conductivity kappas of said honeycomb segment to thermal conductivity kappas of said jointing material for corrugated fibreboard and whose kappa s/kappa a are within the limits of 5-300 and, whose consistency rhoa of said jointing material for corrugated fibreboard is 0.1-4g/cc is offered.

[0009] In this invention, it is desirable that the heat capacity Ha per [ which is expressed with specific heat Cpax consistency rhoa of said jointing material for corrugated fibreboard ] unit volume is in the range of 0.1x106 - 3x106 J/m3 and K. Moreover, it is desirable that a jointing material for corrugated fibreboard contains a metal. Furthermore, as for the honeycomb filter of this invention, it is desirable that two or more jointing materials for corrugated fibreboard with which both [ one side or ] thermal conductivity kappaa and unit volume differ are included, and it is desirable that the range of the coefficient of thermal expansion of a jointing material for corrugated fibreboard is 1x10-6 - 8x10-6/degree C. [ of heat capacity Ha ] [ both ] Moreover, it is desirable that a honeycomb segment uses silicon carbide or silicon-silicon carbide composite material as a principal component. Furthermore, it is desirable that the closure of the opening of the predetermined circulation hole in the honeycomb segment of this invention is carried out in the end face of 1, and the closure of the opening of a residual circulation hole is carried out in other end faces, and it is desirable that the cross section consists of honeycomb segments more than whose 70 capacity %s of a honeycomb filter are 2 900mm 2-10000mm.

[0010]

[Embodiment of the Invention] Hereafter, although the honeycomb filter of this invention is explained to a detail according to a drawing, this invention is not limited to the following operation gestalten. In addition, unless a cross section has a notice especially in below, the perpendicular cross section to the direction of a circulation hole (X shaft orientations) is meant.

[0011] The honeycomb filter 1 of this invention is a honeycomb filter with which it comes to carry out the

junction unification of two or more honeycomb segments 12 which have the circulation hole 3 of a large number penetrated to X shaft orientations divided by the septum 2 through a jointing material for corrugated fibreboard 8, as shown in <u>drawing 1</u> (a), (b), and (c).

[0012] the ratio of thermal conductivity kappas [ as opposed to thermal conductivity kappaa of a jointing material for corrugated fibreboard 8 in the important description of this invention ] of the honeycomb segment 12, i.e., kappa s/kappa a, -- 5-300 -- preferably, it is within the limits of 10-250 still more preferably, and is 8-280, and that 0.1-4g [ /] consistency rhoa [ 0.3-3.5g //cc ] of a jointing material for corrugated fibreboard 8 is in the range of 0.5-3.0g/cc still more preferably preferably cc. By controlling in such range, an extremes-of-temperature rise of a honeycomb filter and/or a temperature gradient can be controlled, and endurance improves. It can consider as the honeycomb filter which could control the maximum temperature and/or temperature gradient at the time of the playback at the time of using especially a honeycomb filter for DPF, and was excellent in endurance.

[0013] For example, when a honeycomb filter is used for DPF, a soot accumulates in a filter, but if the amount of deposition soots increases, the calorific value produced at the time of playback will become large, the maximum temperature to produce and the temperature gradient to generate will increase, and thermal stress will become large. In such a case, in order to control a temperature gradient and to control generating of thermal stress, it is important to control the ratio of thermal conductivity kappas of the honeycomb segment 12 to thermal conductivity kappaa of a jointing material for corrugated fibreboard 8, i.e., a kappa s/kappa a value, rather than to only to control the thermal conductivity of the honeycomb segment 12 or a jointing material for corrugated fibreboard 8. If a kappa s/kappa a value is too small, in order that a jointing material for corrugated fibreboard 8 may not contribute as a thermal break, the inclination for the temperature gradient in a honeycomb segment to become large according to the effectiveness that heat gets across to the next honeycomb segment through a jointing material for corrugated fibreboard 8 arises. On the other hand, to the honeycomb segment 12, if a kappa s/kappa a value is too large, since the thermal conductivity of a jointing material for corrugated fibreboard 8 is too small, the temperature gradient produced in a jointing material for corrugated fibreboard 8 will become large too much, and it will become easy to produce a crack in a jointing material for corrugated fibreboard 8, and will result in breakage of a honeycomb filter depending on the case.

[0014] Moreover, it combines, and since it will not depend on the value of the thermal conductivity of a jointing material for corrugated fibreboard 8 but will be hard coming to contribute a jointing material for corrugated fibreboard 8 as a thermal break if consistency rhoa of a jointing material for corrugated fibreboard 8 is too small, the temperature gradient produced in a segment according to the effectiveness that heat gets across to the next segment through a jointing material for corrugated fibreboard 8 becomes large. On the other hand, if consistency rhoa of a jointing material for corrugated fibreboard 8 is too large, the temperature gradient produced in the jointing-material-for-corrugated-fibreboard 8 interior will become large too much, and it will become easy to produce a crack in a jointing material for corrugated fibreboard 8. Therefore, it can consider as the honeycomb filter excellent in endurance by controlling a kappa s/kappa a value and the value of rhoa in the range of above-mentioned this invention.

[0015] In this invention, thermal conductivity kappas of the honeycomb segment 12 means the thermal conductivity of an average of the septum 2 of the honeycomb segment 12, and a peripheral wall 7, and the circulation hole 3 is not included. Moreover, the ratio of thermal conductivity kappas of the honeycomb segment 12 to thermal conductivity kappaa of a jointing material for corrugated fibreboard 8, i.e., kappa s/kappa a, means the ratio of the average of thermal conductivity kappas of each honeycomb segment 12 in a honeycomb filter 1, and an average of the thermal conductivity of a jointing material for corrugated fibreboard 8.

[0016] Moreover, in this invention, since it will be hard coming to contribute a jointing material for corrugated fibreboard 8 as a thermal break if the heat capacity Ha per unit volume of a jointing material for corrugated fibreboard 8 is too small, heat propagation-comes to be easy to the next honeycomb segment 12 through a jointing material for corrugated fibreboard 8, and it becomes easy to produce the temperature gradient within the honeycomb segment 12. On the other hand, if Ha is too large, the temperature gradient produced in the jointing-material-for-corrugated-fibreboard 8 interior will become easy to become large, and it will become easy to produce a crack in a jointing material for corrugated fibreboard 8. Therefore, it is desirable that it is in the range of  $0.1 \times 106 - 3 \times 106 \text{ J/m3}$  and K, as for the heat capacity Ha per [ which is expressed with specific heat Cpax consistency rhoa of a jointing material for corrugated fibreboard 8 ] unit volume, it is still more desirable that it is in the range of  $0.3 \times 106 - 2.5 \times 106 \text{ J/m3}$  and K, and it is most desirable that it is in the range of  $0.6 \times 106 - 2.0 \times 106 \text{ J/m3}$  and K.

[0017] The value of kappa s/kappa a and the value of rhoa are controllable in the range of this invention by choosing appropriately the quality of the material of a honeycomb segment, the quality of the material of porosity and a jointing material for corrugated fibreboard, etc. As a concrete desirable control means of the value of kappa s/kappa a, and the value of rhoa, a jointing material for corrugated fibreboard considers as the configuration which has a certain set-up pore, and lowering to the value with an eye on the consistency of a jointing material for corrugated fibreboard is mentioned. According to this means, the heat capacity Ha, consistency rhoa, and thermal conductivity kappaa per unit volume can be adjusted in the direction lowered to coincidence. In order for a jointing material for corrugated fibreboard to consider as the configuration which has pore, in case a jointing material for corrugated fibreboard is formed, the approach of adding the ostomy material which contains the hole of the fixed volume beforehand is mentioned to the raw material of a jointing material for corrugated fibreboard. As suitable ostomy material, the empty capsid of various inorganic one, such as balun-like foaming resin and milt balun, and the organic quality of the material etc. is mentioned. Moreover, if it is possible to establish the process of heat treatment after junction, there is also the approach of adding the particle of the starch which forms pore, a cellulose, various inorganic one, and the organic quality of the material as ostomy material by burning down or fusing at predetermined temperature.

[0018] Considering as the configuration whose jointing material for corrugated fibreboard contains metals, such as a metal fiber and a particle, as another desirable control means of a kappa s/kappa a value and the value of rhoa is mentioned. According to this means, thermal conductivity, heat capacity, and a consistency can be adjusted in the direction raised to coincidence. As a desirable metal, copper, stainless steel, etc. are especially mentioned for these fibrous objects etc. preferably. Moreover, it is mentioned that a jointing material for corrugated fibreboard considers as the configuration containing high-specific-gravity material, for example, a silicic acid zirconium, a zirconia, etc. as still more nearly another desirable control means of the value of kappa s/kappa a and the value of rhoa. According to this means, it can adjust in the direction of raising only heat capacity and lowering thermal conductivity.

[0019] By changing the raw material presentation at the time of forming a honeycomb segment, the particle size of a raw material, etc., kappas can be changed and a kappa s/kappa a value can also be made into the suitable range. For example, when porosity can be enlarged, thermal conductivity can be lowered and metal silicon and silicon carbide are used as a component of a honeycomb segment by using an ostomy agent as a raw material of a honeycomb segment, thermal conductivity can be gathered by enlarging the ratio of metal silicon. These means can be chosen suitably or the honeycomb segment of this invention can be suitably manufactured by combining and using.

[0020] Moreover, that two or more jointing materials for corrugated fibreboard with which both [one side or ] thermal conductivity kappaa and unit volume differ are included also has the desirable honeycomb filter of this invention. [ of heat capacity Ha ] [ both ] For example, generation of heat of a center section is in a honeycomb filter especially is large, when using it as DPF. As it follows, for example, is shown in drawing 2, when thermal conductivity kappaa uses high jointing-material-for-corrugated-fibreboard 8B with large heat capacity Ha for jointing-material-for-corrugated-fibreboard 8A with the small heat capacity per unit volume with small thermal conductivity, and the periphery section in the center section, the temperature rise of a center section is controlled and the temperature distribution in a honeycomb filter serve as homogeneity more. Thus, honeycomb segments can be joined using a different jointing material for corrugated fibreboard according to the need property in a filter, and the endurance of a honeycomb filter can be raised more by controlling the temperature gradient in a filter. Two or more jointing materials for corrugated fibreboard with which kappaa differs from Ha are combinable so that there may be especially no limit in this combination and the temperature distribution of the whole honeycomb filter may become homogeneity according to structure, a situation used of a honeycomb filter. The jointing material for corrugated fibreboard with which kappaa differs from Ha can be made with the means which used the above, an ostomy agent, a metal, high-specific-gravity material, etc.

[0021] Since it will be easy to produce a crack in a thermal shock etc. if thermal expansion is large as a jointing material for corrugated fibreboard in the honeycomb filter of this invention, what has a comparatively low coefficient of thermal expansion is desirable. The range of 1x10-6 - 8x10-6/degree C is desirable, the coefficient of thermal expansion in the range of 20 degrees C - 800 degrees C of cement has the still more desirable range which is 1.5x10-6 - 7x10-6/degree C, and its range of 2x10-6 - 6x10-6/degree C is the most desirable. Moreover, it is not desirable in order that thermal stress may concentrate on a joint at the time of heating and cooling, if the difference of the coefficient of thermal expansion of a jointing material for corrugated fibreboard and a honeycomb segment is too large. The difference of the coefficient

of thermal expansion from 20 degrees C to 800 degrees C of a jointing material for corrugated fibreboard and a honeycomb segment is less than [1x10-6/degree C] preferably. A jointing material for corrugated fibreboard is used usually suitably [what used the ceramics as the principal component]. As a raw material for forming a jointing material for corrugated fibreboard, as stated previously, according to a need property, the particle of metals, such as a metal fiber, ostomy material, and various ceramics etc. is used for the mixture of colloidal sols, such as particles, such as aluminum silicate and aluminium phosphate, or fiber and colloidal silica, and a colloidal alumina, for example.

[0022] In this invention, although it is desirable to consist of at least one sort of ingredients chosen from the group which becomes from viewpoints, such as reinforcement and thermal resistance, from cordierite, mullite, alumina, spinel, silicon carbide, and silicon carbide-cordierite system composite material, silicon-silicon carbide system composite material, silicon nitride, lithium aluminium silicate, aluminum titanate, Fe-Cr-aluminum system metals, and such combination as for the principal component of a honeycomb segment, it is thermal conductivity and a heat-resistant point, and especially silicon carbide or silicon-silicon carbide composite material is suitable. Here, a "principal component" means constituting more than 80 mass % still more preferably more than 70 mass % preferably more than 50 mass % of a honeycomb segment. Moreover, in this invention, when a honeycomb segment uses composite material of metal silicon (Si) and silicon carbide (SiC) as a principal component, if there are too few Si contents specified by Si/(Si+SiC) of a honeycomb segment, the effectiveness of Si addition will become is hard to be acquired, and if 50 mass % is exceeded, effectiveness heat-resistant [ which is the description of SiC ], and high temperature conductive will become is hard to be acquired. Therefore, as for Si content, it is desirable that it is five to 50 mass %, and it is still more desirable that it is ten to 40 mass %.

[0023] As for the septum of a honeycomb segment, in this invention, it is desirable that it is the porous body which plays the role of a filter. Although there is especially no limit in the thickness of a septum, if a septum is too thick, the pressure loss at the time of a processed fluid penetrating a porous septum will become large too much, if a septum is too thin, the reinforcement as a filter runs short, and it is not respectively desirable. The range of 30-2000 micrometers of 40-1000 micrometers of thickness of a septum is 50-500 micrometers most preferably still more preferably.

[0024] although there is especially no limit in the cel consistency (the number of the circulation holes per unit cross section) of a honeycomb segment, if a cel consistency is too small in this invention -- the reinforcement as a filter -- and effective -- GSA (geometric surface area) runs short, and if a cel consistency is too large, pressure loss in case a processed fluid flows will become large. a cel consistency -- desirable -- 6-2000 cel / square inch (0.9 - 311 cel / cm2) -- further -- desirable -- 50-1000 cel / square inch (7.8 - 155 cel / cm2) -- it is the range of 100-400 cel / square inch (15.5 - 62.0 cel / cm2) most preferably. Moreover, although there is especially no limit in the cross-section configuration (cel configuration) of a circulation hole, it is desirable that it is either of the viewpoint on manufacture to triangles, squares, hexagons, and corrugated configurations.

[0025] In this invention, although there is no limit in the magnitude of a honeycomb segment, if too small [if each segment is too large, the problem of breakage by thermal stress will arise, and ], the unification by manufacture or junction of each segment becomes complicated and is not desirable. As for the magnitude of a desirable honeycomb segment, it is preferably [900mm 2-10000mm] desirable to 2 and a pan 2 and that are 2 900mm 2-3600mm, and more than 70 capacity % of a honeycomb filter consists of honeycomb segments of this magnitude for the cross section most preferably 900mm 2-5000mm. Although there is especially no limit in the configuration of a honeycomb segment, as shown, for example in drawing 1 (a), the thing whose cross-section configuration has the shape of a square and whose honeycomb segment is the square pole-like can be made into the shape of a basic form, and the configuration of the honeycomb segment by the side of a periphery can be suitably chosen according to the configuration of the honeycomb filter at the time of unifying, as shown in drawing 1 (b) and (c).

[0026] The cross-section configuration of the honeycomb filter of this invention can make especially a limit the shape of a polygon and variant configurations, such as the shape of elliptical besides a circle configuration, a ball-race truck configuration, an ellipse configuration, a trigonum, an abbreviation trigonum, a rectangular head, and an abbreviation square, as there are not, for example, shown in <u>drawing 2</u>. Moreover, although there is especially no limit in the thermal conductivity of the whole honeycomb filter, heat dissipation is too large, and since temperature does not fully rise but regeneration efficiency falls at the time of playback, it is not desirable, even if it is the honeycomb filter of this invention, when thermal conductivity is too high. Moreover, since there is too little heat dissipation when thermal conductivity is too low, a temperature rise is too large and is not desirable. The thermal conductivity in 40 degrees C is 20 - 50

W/mK most preferably 15 to 55 W/mK still more preferably ten to 60 W/mK.

[0027] When using as DPF, as shown in <u>drawing 3</u>, as for especially the honeycomb segment 12 in this invention, it is desirable that the closure of the opening of predetermined circulation hole 3a is carried out in the end face 46 of 1, and the closure of the opening of residual circulation hole 3b is carried out in other end faces 48. As especially shown in <u>drawing 3</u>, it is desirable that while becomes the opposite side mutually and the closure of the adjoining circulation hole 3 is carried out at the end so that end faces 46 and 48 may present the shape of a checker. Thus, by closing, in case the processed fluid which flowed from the end face 46 of 1 passes along a septum 2, it flows out of other end faces 48 and a processed fluid passes along a septum 2 can achieve the duty of a filter and can remove the specified substance.

[0028] Although mentioned as the ceramics which can be used suitable for an above-mentioned honeycomb segment as an ingredient used for the closure, or a metal, one sort or two sorts or more of ingredients chosen from inside can be used suitably.

[0029] When it is going to use the honeycomb filter of this invention for reforming of purification of the exhaust gas of burners, such as heat engines, such as an internal combustion engine, or a boiler, liquid fuel, or gaseous fuel as catalyst support, it is desirable to make the honeycomb filter of this invention support the metal which has a catalyst, for example, catalyst ability. It is desirable for Pt, Pd, and Rh to be mentioned and to make a honeycomb filter support at least one of sorts of these as a metaled typical thing which has catalyst ability.

[0030] The manufacture approach of the honeycomb filter of this invention is explained below. It is used as raw material powder of a honeycomb filter, the above-mentioned suitable ingredient, for example, silicon carbide powder, a binder, for example, methyl cellulose, and hydroxypropoxyl methyl cellulose are added to this, a surfactant and water are added further, and a reversible plastic matter is produced. The honeycomb segment which has a predetermined configuration is obtained by carrying out extrusion molding of this plastic matter. The honeycomb segment of predetermined thermal conductivity kappas is obtained by carrying out heating cleaning in N2 ambient atmosphere, after closing with the ingredient which while becomes the opposite side mutually and the adjoining circulation hole 3 used for manufacture of a honeycomb filter at the end, and the same ingredient and drying further so that an end face may present the shape of a checker after desiccation by microwave and hot blast, and calcinating this in inert atmospheres, such as Ar, after that. After joining the obtained segment using jointing-material-for-corrugated-fibreboard raw materials, such as ceramic cement containing metals, such as for example, an ostomy agent and a metal fiber, high-specific-gravity material, etc., desiccation hardening can be carried out at 200 degrees C, and a honeycomb filter can be obtained.

[0031] Thus, the approach which this contractor usually performs is sufficient as the method of making the manufactured honeycomb filter support a catalyst, for example, it can carry out the wash coat of the catalyst slurry, and can make a catalyst support by drying and calcinating. Moreover, it joins, after making a honeycomb segment support a catalyst, and a catalyst may be made to support, after it is good also as a honeycomb filter and considering as a honeycomb filter.

[0032]

[Example] Hereafter, although this invention is further explained to a detail based on an example, this invention is not limited to these examples.

[0033] (Production of a honeycomb segment) As a raw material, it mixed at SiC powder, metal Si powder, and a mass rate that shows a polymethyl methacrylate in Table 1 as ostomy material, methyl cellulose and hydroxypropoxyl methyl cellulose, a surfactant, and water were added to this, and the reversible plastic matter was produced. Extrusion molding of this plastic matter was carried out, it dried by microwave and hot blast, and the thickness of a septum obtained 380 micrometers, the square about 31.0 cels / cm2 (200 cel / square inch), and whose cross section a cel consistency is 35mm per side, and the honeycomb segment whose die length is 152mm. After while becomes the opposite side mutually and said adjoining circulation hole closed and dried this with the ingredient used for manufacture of a honeycomb filter at the end, and the same ingredient so that an end face might present the shape of a checker, it degreased at about 400 degrees C among the atmospheric-air ambient atmosphere, it calcinated at about 1450 degrees C in Ar inert atmosphere after that, and the segments A and B of the honeycomb filter of the Si association SiC were obtained.

[0034] The porosity, the four-point flexural strength, the Young's modulus, and the thermal conductivity of the honeycomb segments A and B were measured, and the result was also shown in Table 1. Porosity was measured by the Archimedes method. Moreover, thermal conductivity is JIS. Based on the approach of a publication, it measured with the laser flash method to R1611. Four-point flexural strength reaches and

Young's modulus is JIS. It measured by the approach based on R1601. [0035]

[Table 1]

ハニカム セグメント	SiC粉体平均 粒径[μm]	SiC粉末配合 量[質量部]	金属Si平均 粒佳[µm]			造孔材配合 量[質量部]	平均和孔径 [μm]	気孔率 [%]	4点曲げ 強度 [MPa]	ヤング 串 [GPa]	熱伝導率 [W/mK]
Α	50	70	4	30	-	-	15	40	35	25	40
В	32.6	80	4	20	12	20	10	55	12	10	12

[0036] (Preparation of a jointing-material-for-corrugated-fibreboard raw material) By the presentation shown in Table 2, a colloidal silica 40 mass % water solution and clay were mixed as the nature fiber of aluminosilicate with a pitch diameter of 100 micrometers, silicon carbide fine particles with a pitch diameter of 100 micrometers, a silicic acid zirconium, and an inorganic binder, water was added, kneading was performed for 30 minutes using the mixer, and the jointing-material-for-corrugated-fibreboard raw materials 1-5 were prepared. It is here, and the jointing-material-for-corrugated-fibreboard raw materials 2 and 3 add foaming resin as ostomy material, and add Cu fiber with a die length of 1mm as a metal fiber from the jointing-material-for-corrugated-fibreboard raw material 4. Moreover, it changed to silicon carbide, the thing using a silicic acid zirconium was changed to the jointing-material-for-corrugated-fibreboard raw material 6, the nature fiber of aluminosilicate, and silicon carbide, and what was prepared using a silicic acid zirconium and colloidal silica was used as the jointing-material-for-corrugated-fibreboard raw material 7. Thermal conductivity, a consistency, and heat capacity after carrying out desiccation hardening of the jointing-material-for-corrugated-fibreboard raw materials 1-7 at 200 degrees C and considering as jointing materials for corrugated fibreboard 1-7 respectively were measured, and the result was shown in Table 3. Thermal conductivity is JIS. Based on the approach of a publication, it measured with the laser flash method to R1611. Heat capacity measured the consistency for specific heat capacity by measurement and also the Archimedes method with the laser flash method based on JISR1611, and asked for both product as heat capacity.

[0037]

[Table 2]

接合材原料	アルミノシリケート織 稚[質量%]	炭化珪素 [質量%]	建設シルコニウム [質量%]	コロイタ ルシリカ 【賞量%】	粘土[質量%]	水[質量%]	Cu機維 [質量%]	発泡樹脂 [質量%]
1	32	37	_	20	1	10	_	_
2	28	27	-	27	1 ,	9		. 8
3	24	24	_	24	1	8	_	19
4	27	33	_	16	1	8	15	
5	_	69	_	27	1	3	_	_
6	27		46	18	1	8	_	-
7	_	_	50	29	1	20	_	_

[0038]

[Table 3]

接合材	熱伝導率 [W/mK]	密度[g/cc]	熟膨張係数 [×10 <sup>-6</sup> /℃]	熱容量(Ha)×10 <sup>-6</sup> [J/m <sup>3</sup> .K]
1	0.9	1.7	3.2	1.3
2	0.2	1	2.8	0.8
3	0.05	0.6	2.5	0.5
4	2	2	3.8	1.8
5	. 3.5	1.9	3.6	1.3
6	0.3	1.3	3.5	1.4
7	0.1	4.3	3.3	3.1

[0039] (Examples 1-5 and examples 1-3 of a comparison) It used in the combination which shows the honeycomb segments A and B and the jointing-material-for-corrugated-fibreboard raw materials 1-7 which were obtained by the above-mentioned actuation in Table 4, and after joining the honeycomb segment and carrying out desiccation hardening at 200 degrees C, the honeycomb filter of the shape of a with a die length [ the diameter of 144mm and die length of 152mm ] which are shown in drawing 4 cylinder for DPF was obtained by cutting. After having connected the produced honeycomb filter to the exhaust pipe of the 31.

diese'l power plant of direct injection types, operating the engine using the gas oil containing 30 ppm Ce fuel additive made from low DIA and accumulating the soot (soot) of the amount of conventions in a filter, the temperature up of the honeycomb filter was continuously carried out to 600 degrees C with the propane gas burner, the inside of a honeycomb filter was made into 18% of oxygen density by the change of a bypass valve, and the soot was reproduced. Every 2g /of amounts of a soot is increased l. from l. in 4g /, the amount of uptake deposition soots at the time of a crack being accepted in a filter end face in microscope observation was made into the amount of marginal soots, and the result was shown in Table 4. As shown in Table 4, compared with the honeycomb filter obtained in the examples 1-3 of a comparison which the honeycomb filter obtained in the examples 1-5 shows the kappa s/kappa a value and Ha value of this invention, and show the kappa s/kappa a value out of range and Ha value of this invention, it turns out that the value of the amount of marginal soots is clearly excellent greatly in endurance.

[Table 4]

	ハニカムセグメント	接合材	KS/Ka	Ha×10 <sup>-8</sup> [J/m <sup>3</sup> K]	展界スート単積量 【g/リットル】	サンプル外観
実施例1	Α	1	44	0.9	10	マイクロクラック
実施例2	Α	2	200	0.2	12	マイクロクラック
実施例3	Α	5	11	1.9	12	マイクロクラック
実施例4	В	3	240	0.6	12	マイクロクラック
実施例5	В	4	6	1.8	10	マイクロクラック
比較例1	Α	3	800	0.5	8	接合材内クラック
比較例2	В	5	4.3	1.3	8	45度方向クラック
比较例3	Α	7	400	3.1	4	接合材内クラック

[0041] (Examples 6 and 7) As shown in <u>drawing 5</u>, except having considered as the jointing material for corrugated fibreboard of combination as shows the jointing material for corrugated fibreboard of jointing-material-for-corrugated-fibreboard 8A (part of the jointing material for corrugated fibreboard of the shape of a cross joint passing through a core), and jointing-material-for-corrugated-fibreboard 8B (periphery) in Table 3, it was the same approach as an example 1, and the honeycomb filter was created and marginal soot alimentation was measured by the same approach as an example 1. As shown in Table 5, by using a jointing material for corrugated fibreboard with the small value of thermal conductivity and heat capacity for a periphery, temperature distribution became in the direction which is harder to produce, the amount of marginal soots went up by one rank as compared with the examples 1 and 3 using only one kind of jointing material for corrugated fibreboard as a jointing material for corrugated fibreboard, and endurance of a result increased further.

[0042]

[Table 5]

	基材	接合材8A (十字部)	接合材8B (周辺部)	κ Β/ κ a(8A)	κs/κa(8B)	限界スート堆積量 [g/リットル]
実施例6	Α	1	2	44	200	14
実施例7	A	5	2	11	200	14

#### [0043]

[Effect of the Invention] As stated above, since the ratio of thermal conductivity kappas [ as opposed to thermal conductivity kappaa of a jointing material for corrugated fibreboard in the honeycomb filter of this invention ] of said honeycomb segment and kappa s/kappa a were in the range whose consistency rhoa of said jointing material for corrugated fibreboard it is within the limits of 5-300, and is 0.1-4g/cc, they showed good endurance. In addition, although the honeycomb filter of this invention is used especially suitable for DPF, the effectiveness of this invention suppresses an extremes-of-temperature rise of a filter, and is to make the temperature distribution in a filter into homogeneity, and the application is not restricted only to DPF.

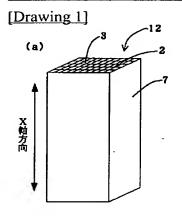
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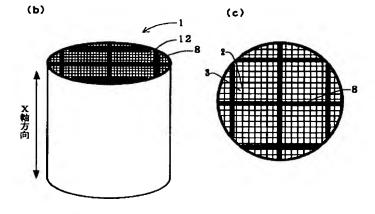
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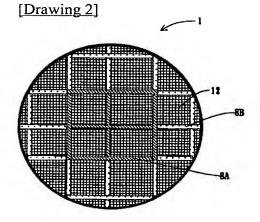
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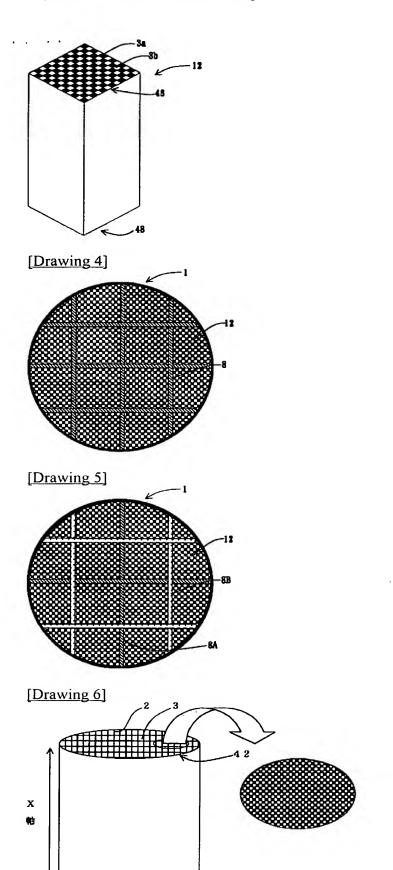
# DRAWINGS .







[Drawing 3]



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### CORRECTION OR AMENDMENT

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[0041]

(Examples 6 and 7)

As shown in drawing 5, except having considered as the jointing material for corrugated fibreboard of combination as shows the jointing material for corrugated fibreboard of jointing-material-for-corrugated-fibreboard 8A (part of the jointing material for corrugated fibreboard of the shape of a cross joint passing through a core), and jointing-material-for-corrugated-fibreboard 8B (periphery) in Table 5, it was the same approach as an example 1, and the honeycomb filter was created and marginal soot alimentation was measured by the same approach as an example 1. As shown in Table 5, by using a jointing material for corrugated fibreboard with the small value of thermal conductivity and heat capacity for a periphery, temperature distribution became in the direction which is harder to produce, the amount of marginal soots went up by one rank as compared with the examples 1 and 3 using only one kind of jointing material for corrugated fibreboard as a jointing material for corrugated fibreboard, and endurance of a result increased further.

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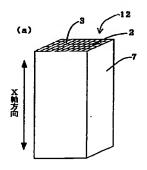
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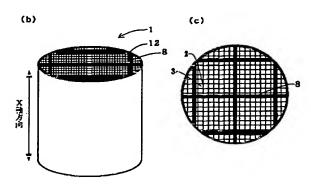
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# (54) 【発明の名称】 ハニカムフィルター

### (57)【要約】

【課題】 温度の過度の上昇を抑制でき、温度分布のばらつきが少なく耐久性に優れたハニカムフィルターを提供する。





## 【特許請求の範囲】

【請求項1】 隔壁により仕切られた、軸方向に貫通する多数の流通孔を有する複数のハニカムセグメントが接合材を介して接合一体化されてなるハニカムフィルターであって、前記接合材の熱伝導率 $\kappa$ aに対する前記ハニカムセグメントの熱伝導率 $\kappa$ sの比、 $\kappa$ s/ $\kappa$ aが5~300の範囲内であって、かつ前記接合材の密度 $\rho$ aが0.1~4g/ccの範囲にあることを特徴とするハニカムフィルター。

【請求項2】 前記接合材の、比熱 $Cpa \times 密度\rhoa$ で表される単位体積当たりの熱容量 $Haが、0.1 \times 10$ 6~ $3 \times 10^6$  J/ $m^3$ ・Kの範囲にあることを特徴とする請求項1に記載のハニカムフィルター。

【請求項3】 接合材が気孔を有することを特徴とする 請求項1又は2に記載のハニカムフィルター。

【請求項4】 接合材が金属を含むことを特徴とする請求項1乃至3の何れか1項に記載のハニカムフィルタ

【請求項5】 熱伝導率 κ a 及び単位体積当たりの熱容量 H a の一方又は両方が異なる 2 以上の接合材を含むことを特徴とする請求項1 乃至4 の何れか1 項に記載のハニカムフィルター。

【請求項6】 接合材の熱膨張率が、 $1 \times 10^{-6} \sim 8 \times 10^{-6}$  / C の範囲であることを特徴とする請求項1 乃至 5 の何れか1 項に記載のハニカムフィルター。

【請求項7】 ハニカムセグメントが、炭化珪素又は珪 素-炭化珪素複合材料を主成分とすることを特徴とする 請求項1乃至6の何れか1項に記載のハニカムフィルタ

【請求項8】 ハニカムセグメントにおける所定の流通 孔の開口部が一の端面において封止され、残余の流通孔 の開口部が他の端面において封止されていることを特徴 とする請求項1乃至7の何れか1項に記載のハニカムフィルター。

【請求項9】 ハニカムフィルターの70容量%以上が、断面積が900mm²~10000mm²であるハニカムセグメントから構成されていることを特徴とする請求項1乃至8の何れか1項に記載のハニカムフィルタ

### 【発明の詳細な説明】

[0001]

【発明の属する技術分野】 本発明は、内燃機関、ボイラー等の排ガス中の微粒子捕集フィルター等に用いられるハニカムフィルターに関し、特に温度の過度の上昇を抑制でき、温度分布のばらつきが少なく耐久性に優れたハニカムフィルターに関する。

[0002]

【従来の技術】 内燃機関、ボイラー等の排ガス中の傲 粒子、特にディーゼル微粒子の捕集フィルター等にハニ カムフィルターが用いられている。

【0003】 この様な目的で使用されるハニカムフィ ルターは、一般に、図6に示すように、隔壁2により仕 切られた、X軸方向に貫通する多数の流通孔3を有し、 端面が市松模様状を呈するように、隣接する流通孔3が 互いに反対側となる一方の端部で封止された構造を有す る。この様な構造を有するハニカムフィルターにおい て、被処理流体は流入口側端面42が封止されていない 流通孔3、即ち流出口側端面44が封止されている流通 孔3に流入し、多孔質の隔壁2を通って隣の流通孔3、 即ち流入口側端面42が封止され、流出口側端面44が 封止されていない流通孔3から排出される。この際隔壁 2がフィルターとなり、例えばディーゼルエンジンから 排出されるスート (スス) などが隔壁に捕捉され隔壁上 に堆積する。この様に使用されるハニカムフィルター は、排気ガスの急激な温度変化や局所的な発熱によって ハニカム構造内の温度分布が不均一となり、ハニカムフ ィルターにクラックを生ずる等の問題があった。特にデ ィーゼルエンジンの排気中の粒子状物質を捕集するフィ ルター(以下DPFという)として用いられる場合に は、溜まったカーボン微粒子を燃焼させて除去し再生す ることが必要であり、この際に局所的な高温化がおこ り、再生温度の不均一化による再生効率の低下及び大き な熱応力によるクラックが発生しやすいという問題があ った。また、再生時の温度分布が均一でないために、フ ィルター全体にわたり最適温度とすることが難しく、再 生効率の向上を図ることが困難であった。

【0004】 このため、ハニカムフィルターを複数に 分割したセグメントを接合材により接合する方法が提案 された。例えば、米国特許第4335783号公報に は、多数のハニカム体を不連続な接合材で接合するハニ カム構造体の製造方法が開示されている。また、特公昭 61-51240号公報には、セラミック材料よりなる ハニカム構造のマトリックスセグメントを押出し成形 し、焼成後その外周部を加工して平滑にした後、その接 合部に焼成後の鉱物組成がマトリックスセグメントと実 質的に同じで、かつ熱膨張率の差が800℃において O. 1%以下となるセラミック接合材を塗布し、焼成す る耐熱衝撃性回転蓄熱式が提案されている。また、19 86年のSAE論文860008には、コージェライト のハニカムセグメントを同じくコージェライトセメント で接合したセラミックハニカム構造体が開示されてい る。さらに特開平8-28246号公報には、ハニカム セラミック部材を少なくとも三次元的に交錯する無機繊 維、無機バインダー、有機バインダー及び無機粒子から なる弾性質シール材で接着したセラミックハニカム構造 体が開示されている。また、熱伝導率の高く、耐熱性の 高い炭化珪素系の材料等を用いてハニカムフィルターを 作ることにより局所的な高温化を防止し、熱応力による ハニカムフィルターの破損を防止することも試みられて いる。

【0005】 しかしながらセグメント化することにより、及び/又は炭化珪素系の材料のように耐熱性の高い材料を用いることにより熱応力による破損はある程度抑制できるものの、ハニカムフィルターの外周部と中心部の温度差を解消することはできず、均一な再生による耐久性の向上という点では不十分であった。また、再生時における局所的な発熱が生じる場合もあった。

【0006】 また、特開2001-162119公報 には、シール材(接合材)層の厚さが0.3~5mmで あって、かつその熱伝導率0.1~10W/mkのセラ ミックフィルタ集合体とすることで、全体の温度を均一 化し部分的な燃え残りが生じにくいフィルターが開示さ れている。しかしながら接合材の厚さと熱伝導率を一定 範囲にすることにより、部分的な燃え残りをなくしスー トの再生効率を上げることはできるものの、局所的に高 温発熱したときに発生する温度勾配を抑制し熱応力を抑 えるには十分ではなく、スート再生可能な限界スート量 の向上という点では不十分であった。また同公報に開示 されているように接合材の厚みを変えることで接合材の 熱伝導率や熱容量を調整することはできるが、接合材の 厚みを増していくとフィルターの有効面積を減少させ、 スート付圧力損失の特性が低下するという別の不具合を 生じるため、低熱伝導率、高熱容量とフィルターの圧力 損失は、接合材の厚みで制御しようとすると背反特性と なり、実際にフィルターに適用可能なシール厚さには限 界がある。

### [0007]

【発明が解決しようとする課題】 本発明はこのような 事情に鑑みてなされたものであり、その目的とするところは、接合材の厚みを特に変えなくても、温度の過度な 上昇を抑制でき、耐久性に優れたハニカムフィルターを 提供することにある。

### [0008]

【課題を解決するための手段】 本発明は、隔壁により仕切られた、軸方向に貫通する多数の流通孔を有する複数のハニカムセグメントが接合材を介して接合一体化されてなるハニカムフィルターであって、前記接合材の熱伝導率 $\kappa$  a に対する前記ハニカムセグメントの熱伝導率 $\kappa$  s の比、 $\kappa$  s  $\kappa$ 

【0009】 本発明において、前記接合材の、比熱C pa×密度 $\rho$ aで表される単位体積当たりの熱容量Haが、 $0.1\times10^6$ ~ $3\times10^6$  J/ $m^3$ ·Kの範囲にあることが好ましい。また、接合材が気孔を有することが好ましく、接合材が金属を含むことが好ましい。さらに、本発明のハニカムフィルターは、熱伝導率 $\kappa$ a及び単位体積当たりの熱容量Haの一方又は両方が異なる2以上の接合材を含むことが好ましく、接合材の熱膨張率

が、1×10<sup>-6</sup>~8×10<sup>-6</sup>/℃の範囲であることが好ましい。また、ハニカムセグメントが、炭化珪素又は珪素一炭化珪素複合材料を主成分とすることが好ましい。さらに、本発明のハニカムセグメントにおける所定の流通孔の開口部が一の端面において封止され、残余の流通孔の開口部が他の端面において封止されていることが好ましく、ハニカムフィルターの70容量%以上が、断面積が900mm²~10000mm²であるハニカムセグメントから構成されていることが好ましい。

# [0010]

【発明の実施の形態】 以下、図面に従って、本発明のハニカムフィルターを詳細に説明するが、本発明は以下の実施形態に限定されるものではない。なお、以下において断面とは、特に断りのない限り流通孔方向(X軸方向)に対する垂直の断面を意味する。

【0011】 本発明のハニカムフィルター1は、例え ば図1(a)、(b)及び(c)に示すように、隔壁2 により仕切られた、X軸方向に貫通する多数の流通孔3 を有する複数のハニカムセグメント12が接合材8を介 して接合一体化されてなるハニカムフィルターである。 【0012】 本発明の重要な特徴は、接合材8の熱伝 導率κaに対するハニカムセグメント12の熱伝導率κ sの比、即ちκs/κaが5~300、好ましくは8~ 280、さらに好ましくは10~250の範囲内であっ T、かつ接合材8の密度 $\rho$  aが0.  $1\sim4$  g/cc、好 ましくは $0.3\sim3.5g/cc$ 、さらに好ましくは 0.5~3.0g/ccの範囲にあることである。この 様な範囲に制御することにより、ハニカムフィルターの 過度の温度上昇及び/又は温度勾配を抑制することがで き耐久性が向上する。特にハニカムフィルターをDPF に用いた際の再生時における最高温度及び/又は温度勾 配を抑制することができ耐久性に優れたハニカムフィル ターとすることができる。

【0013】 例えば、ハニカムフィルターをDPFに 用いた場合、フィルター内にスートが堆積するが、堆積 スート量が増大していくと、再生時に生じる発熱量は大 きくなってゆき、生じる最高温度、発生する温度勾配が 増大し、熱応力が大きくなる。この様な場合に、温度勾 配を制御し、熱応力の発生を抑制するためには、単にハ ニカムセグメント12、あるいは接合材8の熱伝導率を 制御するのではなく、接合材8の熱伝導率καに対する ハニカムセグメント12の熱伝導率κsの比、即ちκs /κa値を制御することが重要である。κs/κa値が 小さすぎると接合材8が断熱層として寄与しないために 接合材8を介して隣のハニカムセグメントに熱が伝わる 効果によりハニカムセグメント内における温度勾配が大 きくなる傾向が生じる。一方で κ s / κ a 値が大きすぎ るとハニカムセグメント12に対して接合材8の熱伝導 率が小さすぎるために接合材8に生じる温度勾配が大き くなりすぎ接合材8にクラックが生じやすくなり、場合

によってはハニカムフィルターの破損に到る。

【0014】 また併せて接合材8の密度 $\rho$ aが小さすぎると接合材8の熱伝導率の値に依らず、接合材8が断熱層として寄与しにくくなるために、接合材8を介して隣のセグメントに熱が伝わる効果によりセグメント内に生じる温度勾配が大きくなる。一方で接合材8の密度 $\rho$ aが大きすぎると接合材8内部に生じる温度勾配が大きくなりすぎ接合材8にクラックが生じやすくなる。従って、 $\kappa$ s $/\kappa$ a値及び $\rho$ aの値を上記本発明の範囲に制御することにより、耐久性に優れたハニカムフィルターとすることができる。

【0015】 本発明において、ハニカムセグメント12の熱伝導率κsとは、ハニカムセグメント12の隔壁2及び外周壁7の平均の熱伝導率を意味し、流通孔3は含まない。また、接合材8の熱伝導率κaに対するハニカムセグメント12の熱伝導率κsの比、即ちκs/κaは、ハニカムフィルター1中の各ハニカムセグメント12の熱伝導率κsの平均と接合材8の熱伝導率の平均との比率を意味する。

【0016】 また、本発明において、接合材8の単位 体積当たりの熱容量Haが小さすぎると接合材8が断熱 層として寄与しにくくなるために接合材8を介して隣の ハニカムセグメント12に熱が伝わりやすくなり、ハニ カムセグメント12内での温度勾配が生じやすくなる。 一方でHaが大きすぎると接合材8内部に生じる温度勾 配が大きくなりやすくなり接合材8にクラックが生じや すくなる。従って、接合材8の、比熱Cpa×密度ρa で表される単位体積当たりの熱容量Haは、0.1×1 06~3×1 06 J/m3・Kの範囲にあることが好まし く、0.3×106~2.5×106J/m³・Kの範囲 にあることがさらに好ましく、O.6×106~2.0 ×106J/m3・Kの範囲にあることが最も好ましい。 【0017】 ハニカムセグメントの材質や気孔率、接 合材の材質等を適切に選択することにより、κs/κa の値及び p a の値を、本発明の範囲に制御することがで きる。 $\kappa s / \kappa$  a の値及び  $\rho$  a の値の具体的な好ましい 制御手段としては、接合材が、ある設定された気孔を有 する構成とし、接合材の密度を狙いとする値に下げるこ とが挙げられる。この手段によれば、単位体積当たりの 熱容量Ηα、密度ρα及び熱伝導率καを同時に下げる 方向に調整することができる。接合材が気孔を有する構 成とするためには、例えば接合材を形成する際に、接合 材の原料に一定体積の空孔を予め含有する造孔材を添加 する方法が挙げられる。好適な造孔材としてはバルーン 状の発泡樹脂、シラスバルーン等の、各種無機、有機材 質の中空粒子などが挙げられる。また接合後に熱処理の 工程を設けることが可能であれば、所定温度で焼失ある いは溶融することによって気孔を形成するデンプン、セ ルロース、各種無機、有機材質の粒子を造孔材として添 加する方法もある。

【0018】 κs/κa値及びρaの値の別の好ましい制御手段としては、接合材が金属繊維、粒子等の金属を含む構成とすることが挙げられる。この手段によれば、熱伝導率、熱容量及び密度を同時に上げる方向に調整することができる。好ましい金属としては銅、ステンレスなど、特に好ましくはこれらの繊維状物等が挙げられる。また、κs/κaの値及びρaの値のさらに別の好ましい制御手段としては、接合材が、高比重材、例えば珪酸ジルコニウム、ジルコニアなどを含む構成とすることが挙げられる。この手段によれば、熱容量のみを上げて熱伝導率を下げるという方向に調整することができる。

【0019】 ハニカムセグメントを形成する際の原料 組成や原料の粒径等を変化させることにより、κsを変化させ、κs/κa値を適切な範囲にすることもできる。例えば、ハニカムセグメントの原料として造孔剤を用いることにより、気孔率を大きくし、熱伝導率を下げることができ、ハニカムセグメントの構成材料として金属珪素と炭化珪素を用いた場合には、金属珪素の比率を大きくすることにより熱伝導率を上げることができる。これらの手段を適宜選択し又は組み合わせて用いることにより本発明のハニカムセグメントを好適に製造することができる。

【0020】 また、本発明のハニカムフィルターが、 熱伝導率 κa及び単位体積当たりの熱容量 Haの一方又 は両方が異なる2以上の接合材を含むことも好ましい。 例えば、DPFとして使用する場合、発熱が大きいのは ハニカムフィルター内の特に中央部である。従って、例 えば図2に示すように、中央部に熱伝導率が小さく単位 体積当たりの熱容量の小さい接合材8A、外周部には熱 伝導率κaが高く熱容量Haの大きい接合材8Bを用い ることにより、中央部の温度上昇が抑制され、ハニカム フィルター内の温度分布がより均一となる。この様に、 フィルター内の必要特性に応じて異なる接合材を用いて ハニカムセグメント同士を接合し、フィルター内の温度 勾配を制御することで、ハニカムフィルターの耐久性を より向上させることができる。この組み合わせに特に制 限はなく、ハニカムフィルターの構造や使用される状況 等に応じて、ハニカムフィルター全体の温度分布が均一 になるように、κa及び/又はHaが異なる複数の接合 材を組み合わせることができる。 κ a 及び/又は H a が 異なる接合材は、上記、造孔剤、金属及び高比重材など を用いた手段で作ることができる。

【0021】 本発明のハニカムフィルターにおける接合材としては熱膨張が大きいと熱衝撃などでクラックを生じやすいために、熱膨張率が比較的低いものが好ましい。接合剤の20 $^{\circ}$  $^$ 

1. 5×10<sup>-6</sup>~7×10<sup>-6</sup>/℃の範囲がさらに好ましく、2×10<sup>-6</sup>~6×10<sup>-6</sup>/℃の範囲が最も好まし

い。また、接合材とハニカムセグメントとの熱膨張係数の差が大きすぎると加熱・冷却時において接合部に熱応力が集中するため好ましくない。接合材とハニカムセグメントとの20℃から800℃までの熱膨張係数の差は、好ましくは1×10-6/℃以下である。接合材は、セラミックスを主成分としたものが通常は好適に用いられる。接合材を形成するための原料としては、例えば珪酸アルミニウム、リン酸アルミニウム等の粒子又は繊維とコロイダルシリカ、コロイダルアルミナ等のコロイダルブルの混合物に、先に述べたように必要特性に応じて金属繊維等の金属、造孔材、各種セラミックスの粒子などが用いられる。

【0022】 本発明において、ハニカムセグメントの 主成分は、強度、耐熱性等の観点から、コージェライ ト、ムライト、アルミナ、スピネル、炭化珪素、炭化珪 素ーコージェライト系複合材料、珪素ー炭化珪素系複合 材料、窒化珪素、リチウムアルミニウムシリケート、チ タン酸アルミニウム、Fe-Cr-Al系金属及びこれ らの組み合わせよりなる群から選ばれる少なくとも1種 の材料からなることが好ましいが、熱伝導率及び耐熱性 の点で、炭化珪素又は珪素-炭化珪素複合材料が特に適 している。ここで、「主成分」とは、ハニカムセグメン トの50質量%以上、好ましくは70質量%以上、さら に好ましくは80質量%以上を構成することを意味す る。また、本発明において、ハニカムセグメントが金属 珪素(Si)と炭化珪素(SiC)の複合材料を主成分 とする場合、ハニカムセグメントのSi/(Si+Si C)で規定されるSi含有量が少なすぎるとSi添加の 効果が得られにくくなり、50質量%を超えるとSiC の特徴である耐熱性、高熱伝導性の効果が得られにくく なる。従ってSi含有量は、5~50質量%であること が好ましく、10~40質量%であることがさらに好ま

【0023】 本発明において、ハニカムセグメントの隔壁は、フィルターの役割を果たす多孔質体であることが好ましい。隔壁の厚さに特に制限はないが、隔壁が厚すぎると多孔質の隔壁を被処理流体が透過する際の圧力損失が大きくなりすぎ、隔壁が薄すぎるとフィルターとしての強度が不足し各々好ましくない。隔壁の厚さは、好ましくは $30\sim2000\mu$ m、さらに好ましくは $40\sim1000\mu$ m、最も好ましくは $50\sim500\mu$ mの範囲である。

【0024】 本発明において、ハニカムセグメントのセル密度(単位断面積当たりの流通孔の数)に特に制限はないが、セル密度が小さすぎると、フィルターとしての強度及び有効GSA(幾何学的表面積)が不足し、セル密度が大きすぎると、被処理流体が流れる場合の圧力損失が大きくなる。セル密度は、好ましくは、6~200セル/平方インチ(0.9~311セル/cm²)、さらに好ましくは50~1000セル/平方イ

ンチ (7.8~155セル/cm²)、最も好ましくは 100~400セル/平方インチ (15.5~62.0 セル/cm²)の範囲である。また、流通孔の断面形状 (セル形状)に特に制限はないが、製作上の観点から、三角形、四角形、六角形及びコルゲート形状のうちのいずれかであることが好ましい。

【0025】 本発明において、ハニカムセグメントの 大きさに制限はないが、各セグメントが大きすぎると、 熱応力による破損の問題が生じ、小さすぎると各セグメ ントの製造や接合による一体化が煩雑となり好ましくな い。好ましいハニカムセグメントの大きさは、断面積が 900mm<sup>2</sup>~10000mm<sup>2</sup>、さらに好ましくは90 Omm<sup>2</sup>~5000mm<sup>2</sup>、最も好ましくは900mm<sup>2</sup> ~3600mm<sup>2</sup>であり、ハニカムフィルターの70容 量%以上が、この大きさのハニカムセグメントから構成 されていることが好ましい。ハニカムセグメントの形状 に特に制限はないが、例えば図1 (a)に示すように断 面形状が四角形状、即ちハニカムセグメントが四角柱状 であるものを基本形状とし、図1(b)、(c)に示す ように一体化した場合のハニカムフィルターの形状に合 わせて外周側のハニカムセグメントの形状を適宜選択す ることができる。

【0026】 本発明のハニカムフィルターの断面形状は特に制限はなく、例えば図2に示すような円形状の他、楕円形状、レーストラック形状、長円形状、三角、略三角、四角、略四角形状などの多角形状や異形形状とすることができる。また、ハニカムフィルター全体の熱伝導率に特に制限はないが、熱伝導率が高すぎると本発明のハニカムフィルターであっても放熱が大きすぎて、再生時に十分に温度が上昇せず再生効率が低下するため好ましくない。また、熱伝導率が低すぎると放熱が少なすぎるために温度上昇が大きすぎて好ましくない。40℃における熱伝導率は好ましくは、10~60W/mK、さらに好ましくは15~55W/mK、最も好ましくは20~50W/mKである。

【0027】 本発明におけるハニカムセグメント12は、特にDPFとして用いる場合には、図3に示すように、所定の流通孔3aの開口部が一の端面46において封止され、残余の流通孔3bの開口部が他の端面48において封止されていることが好ましい。特に、図3に示すように、端面46及び48が市松模様状を呈するように、隣接する流通孔3が互いに反対側となる一方の端部で封止されていることが好ましい。この様に封止することにより、例えば一の端面46から流入した被処理流体は隔壁2を通って、他の端面48から流出し、被処理流体が隔壁2を通る際に隔壁2がフィルターの役目をはたし、目的物を除去することができる。

【0028】 封止に用いる材料としては、上述のハニカムセグメントに好適に用いることができるセラミックス又は金属として挙げたものの中から選択された1種又

は2種以上の材料を好適に用いることができる。

【0029】 本発明のハニカムフィルターを、触媒担体として内燃機関等の熱機関若しくはボイラー等の燃焼装置の排気ガスの浄化、又は液体燃料若しくは気体燃料の改質に用いようとする場合、本発明のハニカムフィルターに触媒、例えば触媒能を有する金属を担持させることが好ましい。触媒能を有する金属の代表的なものとしては、Pt、Pd、Rhが挙げられ、これらのうちの少なくとも1種をハニカムフィルターに担持させることが好ましい。

【0030】 つぎに本発明のハニカムフィルターの製 造方法を説明する。ハニカムフィルターの原料粉末とし て、前述の好適な材料、例えば炭化珪素粉末を使用し、 これにバインダー、例えばメチルセルロース及びヒドロ キシプロポキシルメチルセルロースを添加し、さらに界 面活性剤及び水を添加し、可塑性の坏土を作製する。こ の坏土を押出成形することにより、所定の形状を有する ハニカムセグメントを得る。これを、例えばマイクロ波 及び熱風で乾燥後、端面が市松模様状を呈するように、 隣接する流通孔3が互いに反対側となる一方の端部でハ ニカムフィルターの製造に用いた材料と同様の材料で封 止し、さらに乾燥した後、例えばN₂雰囲気中で加熱脱 脂し、その後Ar等の不活性雰囲気中で焼成することに より所定の熱伝導率κ s のハニカムセグメントを得る。 得られたセグメントを、例えば、造孔剤、金属繊維など の金属、高比重材等を含むセラミックセメント等の接合 材原料を用いて接合した後、200℃で乾燥硬化し、ハ ニカムフィルターを得ることができる。

【0031】 この様にして製造されたハニカムフィルターに触媒を担持させる方法は、当業者が通常行う方法でよく、例えば触媒スラリーをウォッシュコートして乾燥、焼成することにより触媒を担持させることができ

る。また、ハニカムセグメントに触媒を担持させてから 接合し、ハニカムフィルターとしても良く、ハニカムフィルターとしてから触媒を担持させても良い。

#### [0032]

【実施例】 以下、本発明を実施例に基づいてさらに詳細に説明するが、本発明はこれらの実施例に限定されるものではない。

【0033】 (ハニカムセグメントの作製)原料とし て、SiC粉及び金属Si粉、及び造孔材としてポリメ タクリル酸メチルを表 1 に示す質量割合で混合し、これ にメチルセルロース及びヒドロキシプロポキシルメチル セルロース、界面活性剤及び水を添加して、可塑性の坏 土を作製した。この坏土を押出成形し、マイクロ波及び 熱風で乾燥して隔壁の厚さが380μm、セル密度が約 31.0セル/cm²(200セル/平方インチ)、断 面が一辺35mmの正方形、長さが152mmのハニカ ムセグメントを得た。これを、端面が市松模様状を呈す るように、隣接する前記流通孔が互いに反対側となる一 方の端部でハニカムフィルターの製造に用いた材料と同 様の材料で封止して、乾燥させた後、大気雰囲気中約4 00℃で脱脂し、その後Ar不活性雰囲気中で約145 O℃で焼成して、Si結合SiCのハニカムフィルター のセグメントA及びBを得た。

【0034】 ハニカムセグメントA及びBの気孔率、4点曲げ強度、ヤング率及び熱伝導率を測定し、その結果も表1に示した。気孔率はアルキメデス法にて測定した。また、熱伝導率はJIS R1611に記載の方法に準拠してレーザーフラッシュ法にて測定した。4点曲げ強度は及びヤング率は、JIS R1601に準拠した方法にて測定した。

### [0035]

### 【表1】

ハニカムセグメント		SiC粉末配合 量[質量部]	金属Si平均 粒径[µm]	金属Si配合 土[賞主部]	造孔材平均 径[μm]	造孔材配合 量[質量部]	平均細孔径 [μm]	気孔率 [%]	4点曲げ 強度 [MPa]	ヤング 事 [GPa]	殷信導率 [W/mK]
Α	50	70	4	30	-	-	15	40	35	25	40
В	32.8	80	4	20	12	20	10	55	12	10	12

【0036】 (接合材原料の調製)表2に示す組成で、平均径100μmのアルミノシリケート質繊維、平均径100μmの炭化珪素粉体、珪酸ジルコニウム、無機バインダーとしてコロイダルシリカ40質量%水溶液及び粘土を混合、水を加えてミキサーを用いて30分間混練を行い、接合材原料1~5を調製した。ここで接合材原料2、3は造孔材として発泡樹脂を、接合材原料4では金属繊維として長さ1mmのCu繊維を添加したものである。また炭化珪素にかえて珪酸ジルコニウムを用いたものを接合材原料6、アルミノシリケート質繊維及び炭化珪素にかえて、珪酸ジルコニウム及びコロイダル

シリカを用いて調製したものを接合材原料7とした。接合材原料1~7を200℃で乾燥硬化させて、各々接合材1~7とした後の熱伝導率、密度及び熱容量を測定し、その結果を表3に示した。熱伝導率はJIS R1611に記載の方法に準拠してレーザーフラッシュ法にて測定した。熱容量はJISR1611に準拠してレーザーフラッシュ法にて比熱容量を測定、さらにアルキメデス法によって密度を測定し、両者の積を熱容量として求めた。

[0037]

【表2】

接合材原料	アルシンジリケート様 株[質量%]	炭化珪素 [質量%]	建酸ジルコニウム   質量%]	コロイダルシリカ 【質量%】	粘土[質量%]	水[質量%]	Cu接起 [質量%]	発泡樹脂 [質量%]
1	32	37	_	20	1	10	-	-
2	28	27	-	27	1	9	_	. 8
3	24	24	_	24	1	8	_	19
4	27	33	_	16	1	8	15	_
5		69	_	27	1	3	_	
6	27		40	18	1	8		_
7	_	_	50	29	1	20	_	_

[0038]

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接合材	熱伝導率 [W/mK]	密度[g/cc]	熟膨張係数 [×10 <sup>-6</sup> /℃]	热容量(Ha)×10 <sup>-6</sup> [J/m <sup>3</sup> .K]
. 1	0.9	1.7	3.2	1.3
2	0.2	1	2.8	0.8
3	0.05	0.6	2.5	0.5
4	2	2	3.8	1.8
5	3.5	1.9	3.6	1.3
6	0.3	1.3	3.5	1.4
7	0.1	4.3	3.3	3.1

【0039】 (実施例1~5及び比較例1~3)上記の操作により得られたハニカムセグメントA、B及び接合材原料1~7を表4に示す組み合わせで用い、ハニカムセグメントを接合して200℃で乾燥硬化させた後、切削により、図4に示す、直径144mm、長さ152mmのDPF用の円柱状のハニカムフィルターを得た。作製したハニカムフィルターを、直噴式3リットルディーゼルエンジンの排気管に接続し、30ppmのローディア社製Ce燃料添加剤を含有する軽油を用いてエンジンを運転し、規定量のスート(スス)をフィルターに溜めた後、続けてプロバンガスバーナーにてハニカムフィルターを600℃に昇温させ、バイパスバルブの切り替えによりハニカムフィルター内を18%の酸素濃度とし

スートを再生した。スートの量を4g/リットルから2g/リットルずつ増やしていき、顕微鏡観察においてフィルター端面にクラックが認められた時点の捕集堆積スート量を限界スート量とし、その結果を表4に示した。表4に示すように、実施例1~5で得られたハニカムフィルターは、本発明のκs/κa値及びHa値を示すものであり、本発明の範囲外のκs/κa値及びHa値を示す比較例1~3で得られたハニカムフィルターに比べて、限界スート量の値が大きく耐久性において明らかに優れていることがわかる。

[0040]

【表4】

	ハニカムセグメント	接合材	KS/KZ	He×10 <sup>-4</sup> [J/m <sup>3</sup> K]	限界メート堆積量 【g/リットル】	サンプル外観
実施例1	А	1	44	0.9	10	マイクロクラック
実施例2	Α	2	200	0.2	12	マイクロクラック
実施例3	Α	5	11	1.9	12	マイクロクラック
実施例4	В	3	240	0.6	12	マイクロクラック
実施例5	В	4	6	1.8	10	マイクロクラック
比较例1	Λ	3	800	0.5	8	接合材内クラック
比较例2	В	5	4.3	1.3	6	45度方向シラック
比較例3	Α	7	400	3.1	4	接合材内シラック

【0041】 (実施例6、7) 図5に示すように、接合材8A(中心を通る十字状の接合材の部分)と接合材8B(周辺部)の接合材を表3に示すような組み合わせの接合材とした以外は実施例1と同様の方法で、ハニカムフィルターを作成し、実施例1と同様の方法で限界スート堆積量を測定した。結果は、表5に示すように、周辺部に熱伝導率、熱容量の値の小さい接合材を用いるこ

とにより、温度分布がより生じにくい方向になり、接合材として1種類の接合材のみを用いた実施例1、3と比較して限界スート量が1ランク上がり、さらに耐久性が増した。

[0042]

【表5】

	基材	投合材8A (十字部)	接合材8B (周辺部)	K8/Ka(8A)	кэ/ка(8В)	限界スート堆積量 【g/リットル】
実施例8	A	1	2	44	200	14
実施例7	_ A _	5	2	11	200	14

### [0043]

【発明の効果】 以上述べてきたように本発明のハニカムフィルターは、接合材の熱伝導率 $\kappa$ aに対する前記ハニカムセグメントの熱伝導率 $\kappa$ sの比、 $\kappa$ s/ $\kappa$ aが5~300の範囲内であって、かつ前記接合材の密度 $\rho$ aが0.1~4g/ccの範囲にあるので、良好な耐久性を示した。なお、本発明のハニカムフィルターはDPFに特に好適に用いられるが、本発明の効果は、フィルターの過度の温度上昇を抑え、フィルター内の温度分布を均一にすることにあり、その用途はDPFだけには限られない。

### 【図面の簡単な説明】

【図1】 (a) は本発明に係るハニカムセグメントの一形態を示す模式的な斜視図、(b) は、本発明のフィルターの一形態を示す模式的な斜視図、(c) は本発明のハニカムフィルターの一形態を示す模式的な平面図で

ある。

【図2】 本発明のハニカムフィルターの別の形態を示す模式的な平面図である。

【図3】 本発明に係るハニカムセグメントの別の実施 形態を示す模式的な斜視図である。

【図4】 実施例1~5において作成された本発明のハ ニカムフィルターを示す模式的な平面図である。

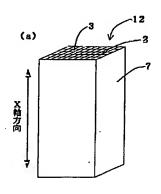
【図5】 実施例6、7において作成された本発明のハニカムフィルターを示す模式的な平面図である。

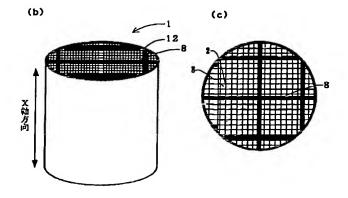
【図6】 従来のハニカムフィルターを示す模式的な斜視図である。

### 【符号の説明】

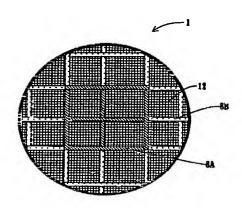
1…ハニカムフィルター、2…隔壁、3、3a、3b… 流通孔、7…外周壁、8、8A、8B…接合材、12… ハニカムセグメント、42…流入口側端面、44…流出 口側端面、46、48…端面。

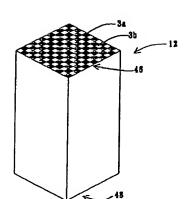
【図1】



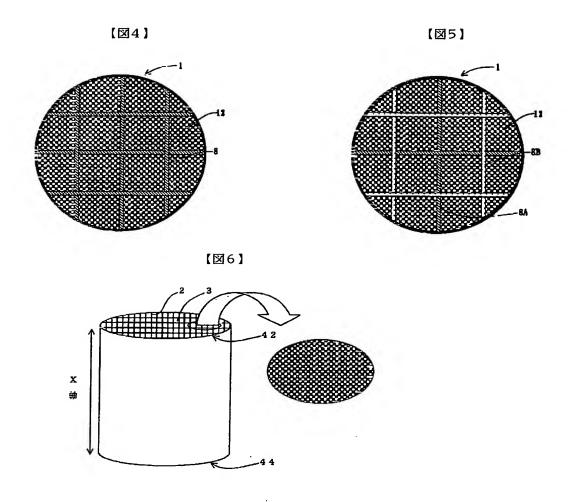


【図2】





【図3】



フロントページの続き

Fターム(参考) 3G090 AA02 AA03 BA01 CA04 4D019 AA01 BA05 BB06 BD10 CA01 CB10